UNCLASSIFIED

AD 407 274

DEFENSE DOCUMENTATION CENTER
FOR
SCIENTIFIC AND TECHNICAL INFORMATION
CAMERON STATION, ALEXANDRIA, VIRGINIA

UNCLASSIFIED

NOTICE: When government or other drawings, specifications or other data are used for any purpose other than in connection with a definitely related government procurement operation, the U. S. Government thereby incurs no responsibility, nor any obligation whatsoever; and the fact that the Government may have formulated, furnished, or in any way supplied the said drawings, specifications, or other data is not to be regarded by implication or otherwise as in any manner licensing the holder or any other person or corporation, or conveying any rights or permission to manufacture, use or sell any patented invention that may in any way be related thereto.

63-4-1

CATALOGED BY DDC
AS AD No. 407274

407 274

TR-1028

STATISTICAL EVALUATION OF CALORIMETRIC MEASUREMENTS ON HEAT-SOURCE MATERIALS

Sheldon G. Levin
Robert E. McIntyre

DDC
RECEIVED
JUL 8 1963
TISIA A

28 March 1963

HARRY DIAMOND LABORATORIES
FORMERLY: DIAMOND ORDNANCE FUZE LABORATORIES
ARMY MATERIEL COMMAND
WASHINGTON 25, D.C.

HARRY DIAMOND LABORATORIES

Robert W. McEvoy
LtCol, Ord Corps
Commanding

B. M. Horton
Technical Director

MISSION

The mission of the Harry Diamond Laboratories is:

(1) To perform research and engineering on systems for detecting, locating, and evaluating targets; for accomplishing safing, arming, and munition control functions; and for providing initiation signals: these systems include, but are not limited to, radio and non-radio proximity fuzes, predictor-computer fuzes, electronic timers; electrically-initiated fuzes, and related items.

(2) To perform research and engineering in fluid amplification and fluid-actuated control systems.

(3) To perform research and engineering in instrumentation and measurement in support of the above.

(4) To perform research and engineering in order to achieve maximum immunity of systems to adverse influences, including countermeasures, nuclear radiation, battlefield conditions, and high-altitude and space environments.

(5) To perform research and engineering on materials, components, and subsystems in support of above.

(6) To conduct basic research in the physical sciences in support of the above.

(7) To provide consultative services to other Government agencies when requested.

(8) To carry out special projects lying within installation competence upon approval by the Director of Research and Development, Army Materiel Command.

(9) To maintain a high degree of competence in the application of the physical sciences to the solution of military problems.

The findings in this report are not to be construed as an official Department of the Army position.

UNITED STATES ARMY MATERIEL COMMAND
HARRY DIAMOND LABORATORIES
WASHINGTON 25, D.C.

TR-1028

OMS Code 4070.17.30021
HDL Proj 75333

28 March 1963

STATISTICAL EVALUATION OF CALORIMETRIC MEASUREMENTS ON HEAT-SOURCE MATERIALS

Sheldon G. Levin
Robert E. McIntyre

FOR THE COMMANDER:
Approved by

P.E. Landis
Chief, Laboratory 900

Qualified requesters may obtain copies of this report from ASTIA.

CONTENTS

LIST OF TABLES

ABSTRACT

A statistical study was made to determine the precision of calorimetric determinations made on the Parr Model 1411 calorimeter. In addition, the calorific values for continuous-production heat-source material and for an experimentally prepared homogeneous material were determined, ~~at Harry Diamond Laboratories (HDL) and by a contractor,~~ and the variability of measurements are compared. It was found that operator-skill and techniques employed can result in a significant increase in the precision of measurements obtained. ~~HDL~~ procedures intended to increase precision are given.

When the determinations with the Parr Model 1411 calorimeter are carefully made, the variance in calorific values due to the calorimetry is small compared to the variance of the determinations made on samples from a typical run of continuous-production heat-source material.

1. INTRODUCTION

In the most common type of calorimeter, a chemical reaction proceeds rapidly to completion in a bomb surrounded by a known quantity of water in an insulated container, and the rise in temperature of the water is measured with a sensitive thermometer. Then, the heat evolved is calculated as a product of the temperature rise and the total heat capacity of the water and calorimeter. The heat capacity of the calorimeter is obtained either by measuring a reaction of known heat evolution or by introducing a known quantity of heat.

In prior work at HDL, the electrically ignited peroxide bomb calorimeter, Model 1401, manufactured by the Parr Instrument Company, had been found not sufficiently precise for use in the measurement of the heat evolved in burning thermite-type heat-source materials (ref 1). Acceptance of the material is based primarily upon calorimetric results. Accurate and precise measurements are necessary because heat-source materials are expensive, and the unnecessary rejection of material must be minimized.

The Parr Model 1401 calorimeter had been modified specifically for use as a quality control instrument in the heat-source industry (ref 1), and the modified model is called the Model 1411 calorimeter. This instrument and a Parr procedure were studied at HDL (ref 2) and have been employed in the heat-source industry for two years. The accuracy of the Model 1411 calorimeter and the precision of measurements made with it had not been evaluated statistically, and one aim of this report is to provide such an evaluation. An equally important objective of this report is to examine the nature and magnitude of fluctuations in calorific measurements of continuous-production heat-source material. A major difficulty in determining the precision of the instrument is due to the large number of steps involved in the operation; i.e., it is difficult to distinguish between the variability due to operator-skill and that inherent in the measuring process.

The contractor employed for this study showed the ability and indicated the willingness to perform the required calorimetry in the allotted time. On the basis of data from quality control sampling, HDL believed that the contractor's skill was typical of the organizations engaged in the production and testing of pyrotechnic materials. His cooperation in the study was enlisted so that the quality of the calorimetry reflected the usual level of performance.

The comparisons made in this report are not intended to reflect on the contractor's work or of the operator's skill. Instead the statements are interpretations of statistical significance tests and assignments of variation to categories thought to be meaningful.

The period covered by the study described in this report was 10 May 1961 to 5 September 1961. Improvements made at the contractor's plant as a result of insights gained during and after the study are not described.

For this study, an HDL technician was trained in calorimetry through about forty practice runs under close supervision using the Parr Model 1411 calorimeter and the Parr procedure with modifications that were introduced to improve the precision of measurements. The modifications are as follows: The thermometer used to measure the temperature rise was tapped with an electromechanical vibrator (ordinary doorbell vibrator) mounted on the top of the calorimeter, to prevent sticking of the mercury column. (This operation was formerly carried out by tapping the thermometer with a pencil.) Extreme care was taken to clean all bomb parts between runs, and the Dewar flask containing the bomb and water was wiped completely dry after each cleaning. A lucite ring that centered the bomb in the Dewar flask was removed each time, to insure thorough cleaning. When weighing the water, it could not be assumed that the weight of the Dewar flask remained constant from run to run; it had to be brought to balance each time with a tare before weighing the water. Temperature readings were taken as close to prescribed times as possible because a 10-sec error altered the radiation correction.

The results obtained by HDL do not represent an extreme that might be expected of the calorimeter because test conditions were no better than average for this type of work. However, the precision of measurements was possibly improved in using the modified procedure.

2. PRECISION AND ACCURACY OF MEASUREMENTS

Five practice calibration runs were made with benzoic acid and oxygen, and then ten calibration runs were made with the same materials. Results are given in Table I.

Because of some limitations in the calibration procedure using benzoic acid, which are discussed in section 2.1, a homogeneous heat powder was prepared at HDL for parallel determinations. From this material, samples of about 3 g were taken. Twenty determinations of its calorific

TABLE I. CALORIMETRIC DETERMINATION OF BENZOIC ACID

(Calibration runs by HDL)

Calibration		Calorific Value	
Sample weight (g)	Water equivalent (cal/°C)	Heat* produced (cal)	Calorific* value (D**) (cal/g)
0.1474	515.61	931.4	6319.0
0.1724	516.26	1089.2	6318.8
0.1779	515.79	1122.8	6311.1
0.1664	515.73	1053.6	6332.0
0.1519	515.64	1149.1	6316.9
0.1803	515.69	1138.2	6312.9
0.1925	514.56	1216.1	6317.4
0.1945	516.11	1228.5	6316.0
0.1930	515.88	1219.7	6319.7
0.1872	515.77	1182.6	6317.2

$\overline{W} = 515.7$ cal/°C $\overline{D}^{**} = 6318.0$

$\hat{\sigma}^2_W = 0.2043$ $\hat{\sigma}^2_{D^{**}} = 31.009$

*Values calculated using the average water equivalent $\overline{W}$ of 515.7 cal/°C and treating the calorific value of benzoic acid as unknown.

value were made at HDL, and forty determinations were made in the contractor's laboratory. The data obtained for this material are listed in Tables II and III.

With the data taken, a statistical evaluation was made to determine the precision of the measurement. Precision refers to the agreement among repeated measurements; by convention, it is taken to be the reciprocal of the standard deviation of independent repeated measurements on the same instrument. The sample deviation $\hat{\sigma}$ is a measure of imprecision since it increases as precision decreases. The square of the standard deviation $\hat{\sigma}^2$ is called the variance and is a measure of imprecision that is useful in making comparisons of the imprecision of two sets of data.

The following additional symbols will be used in evaluating data. The symbols $\overline{C}$ and $\overline{D}$ designate the average of a series of determinations by the contractor and by HDL, respectively; C and D used as subscripts designate contractor and HDL data, respectively; one and two asterisks

TABLE [illegible] CALORIMETRIC DETERMINATION OF HOMOGENEOUS HEAT-POWDER SAMPLES AT HDL.

Sample weight (g)	Total calorific value (cal)	Calorific value per unit weight (cal/g)
[illegible]	[illegible]	342.61
3.9749	1363.41	342.65
3.9720	1361.35	342.74
3.9723	1362.90	343.10
3.9802	1363.41	342.55
3.9929	1368.05	342.62
3.9749	1362.38	342.75
3.9644	1358.77	342.74
3.9943	1367.02	342.24
3.9859	1367.02	342.96
3.9709	1361.35	342.83
3.9869	1368.95	342.36
3.9861	1364.44	342.30
3.9805	1365.89	343.16
3.9757	1362.38	342.68
3.9768	1362.90	342.54
3.9810	1363.93	342.61
3.9903	1367.02	342.59
3.9864	1366.51	342.79
3.9854	1363.41	342.10

Water equivalent = [illegible]

Average $\bar{D}^{*}$ = 342.645 cal/g

Variance $\hat{\sigma}^2_{D^*}$ = 0.071

TABLE III. CALORIMETRIC DETERMINATION OF HOMOGENEOUS HEAT-POWDER SAMPLES BY CONTRACTOR

Series No. 1			Series No. 2		
Sample weight (g)	Total calorific value (cal)	Calorific value per unit weight (cal/g)	Sample weight (g)	Total calorific value (cal)	Calorific value per unit weight (cal/g)
3.0223	1050.81	347.69	2.5882	900.69	348.00
2.8749	999.17	347.55	2.5638	897.56	350.09
2.9175	1016.74	348.50	2.8938	1004.30	347.05
3.0609	1068.91	349.22	2.9382	1017.84	346.42
3.0206	1050.81	347.88	2.9060	1004.82	345.78
2.9504	1028.99	348.76	2.9199	1011.07	346.27
2.9004	1008.22	347.62	2.9324	1028.78	350.83
2.9085	1011.42	347.75	2.9133	1017.84	349.38
3.0173	1050.81	348.26	3.0935	1073.55	347.04
3.0237	1054.01	348.58	2.9446	1028.78	349.38
3.0327	1054.54	347.72	2.9155	1011.07	346.79
2.9817	1039.63	348.67	2.8755	999.62	347.63
2.9595	1038.57	350.93	3.1148	1090.22	350.01
2.9237	1019.40	348.67	2.9536	1025.65	347.25
2.9449	1025.79	348.33	3.0156	1048.04	347.54
3.0010	1057.20	352.28	3.1404	1094.90	348.65
2.9137	1013.02	347.67	3.0467	1074.07	352.54
2.9545	1033.78	349.90	3.0066	1038.15	345.29
2.9494	1029.52	349.06	3.0994	1075.64	347.05
2.9686	1026.32	345.73	3.0818	1063.66	345.14

$\bar{C}^*_1 = 348.538$ $\hat{\sigma}^2_{C^*_1} = 1.869$ $\bar{C}^*_2 = 347.906$ $\hat{\sigma}^2_{C^*_2} = 3.801$

designate homogeneous-heat-powder and benzoic-acid calibration data, respectively; and μ designates the average of an infinite number of measurements; i.e., the population mean.

2.1 Special Considerations in Using Benzoic Acid in the Calorimeter

Benzoic acid is usually used to calibrate the calorimeter. Since pure benzoic acid is a homogeneous material, it is unimportant that it is destroyed in a determination and that real repeat measurements cannot be made. The benzoic acid calibration does present some special problems, primarily those associated with the requirement that it be burned in an oxygen atmosphere rather than in argon, as is the case with the thermite-type mixtures. When using benzoic acid, a platinum capsule is attached inside the bomb to hold the pellet, and a platinum rather than nichrome fuze wire is used to ignite the mixture; the USP grade oxygen is admitted at between 30 and 35 atmospheres.

A small residue of carbon is found on the capsule in many runs, and it is difficult to decide what level of residue indicates incomplete combustion, hence, whether the runs should be discarded or recorded. Since a residue always results in a high water equivalent, it always results in a directed error that can lead to a bias in the average.

Since benzoic acid supplied by the National Bureau of Standards is in crystal form, and some is likely to be blown away when the oxygen is introduced, the Parr Instrument Company has made pelletized benzoic acid available. The weight of the individual pellets varies considerably, but the calorific value is specified to better than one part in 10,000. Since the calorific value is very high and the oxygen available is limited, the weight of the sample benzoic acid pellets is limited to less than 0.200 g.

2.2 Comparison of Calorimetric Determinations on Benzoic Acid and Homogeneous Heat Powder

Table I presents the water equivalent values computed for the ten HDL determinations using standard benzoic acid pellets weighing between 0.1474 and 0.1945 g and having a known heat of combustion of 6318 cal/g. In addition to the water equivalent values, the calorific values of benzoic acid calculated using the average water equivalent $\bar{W}$ of 515.7 cal/°C are given. The latter values were computed, so that the variances obtained with benzoic acid and with the homogeneous heat powder could be compared in terms of calorific value. Table II presents the calorimetric data obtained at HDL on twenty samples of the homogeneous heat powder. The samples weighed between 3.9689 and 3.9943 g.

Using the Parr 1411 calorimeter in its recommended range of 800 to 1500 cal, the errors in evaluating the total calories per sample are relatively constant. Since the heat of combustion of the benzoic acid is 6318 cal/g and of the homogeneous heat powder is about 342 cal/g, sample weights as given above are required to evolve the total calories in the recommended range. A 0.0001-g error in weighing would be one part in 1800 for the benzoic acid and one part in 40,000 for the homogeneous heat powder. If the total calories were the same for each, then the resulting ratio of errors is,

$$\frac{1/1800}{1/40,000} = 22:1$$

To statistically compare the variances in calorific value of benzoic acid and homogeneous heat powder, the data in Tables I and II are used to form the ratio,

$$F = \frac{\text{Variance of HDL benzoic acid runs}}{\text{Variance of HDL homogeneous heat power runs}}$$

$$\frac{\hat{\sigma}^2_{D^{**}}}{\hat{\sigma}^2_{D^*}} = \frac{31.009}{0.071} = 436.72$$

A. H. Bowker and G. J. Lieberman (ref 3) show that the critical value for the F ratio at the 1 percent significance level is 3.52, which is grossly exceeded in this case. This result is to be expected since the ratio of the two errors is 22:1.

Although the statistical test showed that the ratio of the variances is significant, the result is misleading, since the weights of the two materials were so disparate. Examination of another and perhaps more appropriate measure of spread will shed additional light on the comparison. The coefficients of variation give "normalized" standard deviations. If the variability is proportional to the average, then dividing the standard deviation by the average to obtain the percent variation (also called the coefficient of variation) provides a unitless measure of spread that will tend to remove the effect of the weights. This measure will allow the comparison of the variability of the benzoic acid determinations with the variability of the homogeneous-heat-powder calorific determinations.

$$\frac{\hat{\sigma}_{\bar{D}^*}}{\bar{D}^*} = \frac{\sqrt{0.071}}{342.646} = 0.00078$$

or approximately 0.08 percent

$$\frac{\hat{\sigma}_{D^{**}}}{\bar{D}^{**}} = \frac{\sqrt{31.009}}{6318.0} = 0.00088$$

or approximately 0.09 percent.

Since the coefficients of variation are so close, it must be assumed that the lesser sample weight results in the larger variation in calories per gram for the benzoic acid determinations. It can be concluded that on the basis of percent variation, the two materials provide an equally good estimate of the instrument's precision.

2.3 Comparison of Variance of Contractor's Calorimetry with that of HDL

Table III presents the calorimetric data obtained by a contractor using forty samples of the same batch of homogeneous heat powder as that used in the HDL determinations of Table II. To compare the variability of the two sets of data, form the ratio:

$$F = \hat{\sigma}^2_{C*}/\hat{\sigma}^2_{D*} = 2.86/0.071 = 40.28$$

where $\hat{\sigma}^2_{C*}$ is the variance of all forty runs by the contractor. The contractor made forty runs and HDL made twenty, so that the critical value of F is 2.76 (ref 3) at the 1-percent significance level; the computed ratio is therefore highly significant. Thus the contractor's measurements are more variable than HDL's and his precision is lower.

2.4 Determination of the Accuracy of Contractor's Measurements

The homogeneous heat powder cannot serve as a check on the accuracy of calorimetric determinations unless its calorific value has first been carefully determined in a calorimeter of known accuracy. However, the HDL calorimeter had been recently calibrated using benzoic acid, and had previously been checked against the calorimeter at the National Bureau of Standards (ref 1). Therefore, if a significant difference exists between the means of data obtained by the contractor and by HDL, it will be assumed that the contractor's data are in error.

To evaluate the accuracy of the contractor's calibration using homogeneous heat powder, it is necessary to test $\mu_{C*} = \mu_{D*}$ against $\mu_{C*} \neq \mu_{D*}$. It has been shown previously that the variances are unequal; hence, the degrees of freedom are appropriately adjusted. The procedure of adjustment is given in reference 3. "Student's" t is computed as follows:

$$t = \frac{\bar{C}* - \bar{D}*}{\sqrt{\frac{\hat{\sigma}^2_{C*}}{40} + \frac{\hat{\sigma}^2_{D*}}{20}}} = \frac{348.222 - 342.646}{\sqrt{\frac{2.860}{40} + \frac{0.071}{20}}} = \frac{5.576}{.2739} = 20.36$$

The adjusted degrees of freedom are 43, and the associated critical value of t at the 99 percent confidence level (ref 3) is: $t_{.01} = 2.71$. This value is far exceeded by the computed value. The inference is, therefore, that the means are not equal and that the contractor's calorimetry shows an estimated bias of 348.222 - 342.646 = + 5.6 cal/g.

2.5 Expressions of Imprecision

Using statistics, section 2 has presented comparisons of calorimetric measurements of benzoic acid and of homogeneous heat powder made at HDL. It has also made comparisons of HDL measurements with a contractor's measurements made under similar conditions. These statistics and others derived from them can be used to obtain measures of uncertainty or imprecision of the measurements themselves. Some of these measures which are in the summary of statistics, Table IV, will be briefly explained.

The percent variation, $\hat{\sigma}/\overline{X}$, also called the coefficient of variation is a unitless measure of dispersion that can be used to compare the variability of quantities when the variability is related to the mean. This quantity or the ordinary sample standard deviation, $\hat{\sigma}$, is most often used as a measure of imprecision of individual measurements.

The standard error (S.E. = $\hat{\sigma}/\sqrt{n}$) of the mean provides an estimate of the standard deviation of the sample means and an estimate of imprecision for the average of a set of observations. The tolerance limits assume that the individual observations are normally distributed and the ones given below provide limits within which one would expect to capture 95 percent of all such measurements with 90 percent confidence. Although tolerance limits are not a commonly used measure of imprecision of individual observations, they are a valid measure and are included, since they have been used in previous reports on this subject (ref 1, 2 and 4).

TABLE IV. SUMMARY STATISTICS OF BENZOIC ACID AND HOMOGENEOUS HEAT-POWDER DETERMINATIONS

HDL Benzoic Acid Measurements

Water Equivalent	Derived Calorific Value
$\bar{W} = 515.70$ cal/°C	$\bar{D}^{**} = 6318.0$ cal/g
$\hat{\sigma}^2_W = 0.2043$	$\hat{\sigma}^2_{D^{**}} = 31.009$
$\hat{\sigma}_W = 0.452$ cal/°C	$\hat{\sigma}_{D^{**}} = 5.57$ cal/g
% variation $= 100 \frac{\hat{\sigma}_W}{\bar{W}} = 0.088\%$	$100 \frac{\hat{\sigma}_{D^{**}}}{\bar{D}^{**}} = 0.088\%$
S.E. of mean $\hat{\sigma}_W/\sqrt{10} = 0.143$ cal/°C	$\hat{\sigma}_{D^{**}}/\sqrt{10} = 1.760$ cal/°C
95% tolerance limits) (90% confidence level) 515.70 ± 1.36 cal/°C	6318.0 ± 16.8 cal/g

HDL Homogeneous Heat-Powder Measurements	Contractor Homogeneous Heat Powder Measurements
$\bar{D}^* = 342.646$ cal/g	$\bar{C}^* = 348.222$ cal/g
$\hat{\sigma}^2_{D^*} = 0.0710$	$\hat{\sigma}^2_{C^*} = 2.860$
$\hat{\sigma}_{D^*} = 0.267$ cal/g	$\hat{\sigma}_{C^*} = 1.691$ cal/g
% variation $= 100 \frac{\hat{\sigma}_{D^*}}{\bar{D}^*} = 0.078\%$	$100 \frac{\hat{\sigma}_{C^*}}{\bar{C}^*} = 0.486\%$
S.E. of mean $= \hat{\sigma}_{D^*}/\sqrt{20} = 0.0597$ cal/g	$\hat{\sigma}_{C^*}/\sqrt{40} = 0.267$ cal/g
95% tolerance limits) (90% confidence level) = 342.65 ± 0.69 cal/g	348.22 ± 3.95 cal/g

3. EVALUATION OF THE MAGNITUDE AND NATURE OF VARIABILITY IN HEAT-SOURCE MATERIAL

Samples of continuous-production heat-source material are taken in a manner designed to assure that they represent normal production. In practice, the producer takes a gross sample at 30-ft intervals, divides the sample into four consecutive sections, each containing sufficient material for a single calorimetric determination, and then picks one of these sections at random for his regular quality control tests. For purposes of the present work, the contractor was requested to select and test the material as usual, and to send the balance of all gross samples from a week's production to HDL. HDL then selected twenty of these three-section samples from one production run for this study, and withdrew the two sections that were adjacent to each other in each sample. The remaining section of each sample, which had been adjacent to the one originally withdrawn by the contractor, was identified by code number and returned to the contractor. Thus, both HDL and the contractor were able to analyze adjacent sections each time, although the relative position of the adjacent sections within the gross sample varied. Although the twenty samples were chosen from a single run of material, the code numbers were deliberately chosen to suggest that all the production runs were represented. This was done to make it unlikely that the contractor could have identified the samples returned by HDL with the single production run from which they came and hence to insure the validity of the scrutiny test described in 3.3.

Calorimetric determinations were then made on the continuous-production heat-source material. Data obtained by HDL and the contractor are presented in figure 1 and Table V. In figure 1, calorific value is plotted against the position of the gross sample in the production run (I.D. Number). The contractor's first series is his normal sampling, while his second series is the coded one sent to him by HDL. The order in which the HDL determinations were made was randomized since no comparison of the two series was required.

Summarized in Tables VI and VII are all the statistics that are required to perform the statistical tests described herein. A preliminary evaluation of the frequency distributions from which these statistics were computed showed that the underlying distributions can be considered Gaussian except in the case of the contractor's measurements on the continuous-production heat-source material. Rather than resort to techniques whose interpretation is not straightforward, the standard variance ratio test will be used, and a conservative approach will be employed when testing for significance.

In this section, $\hat{\sigma}$, $\hat{\sigma}^2$, and μ are as previously defined in section 2; one asterisk again indicates homogeneous heat-powder calibration data, and the subscripts C and D again indicate the contractor and HDL data, respectively. $\overline{C}$ and $\overline{D}$ refer to the sample average; thus, $\overline{D}$ is the average of the HDL measurements on continuous-production heat-source material and is an estimate of the mean μ_D of the population from which the sample is drawn.

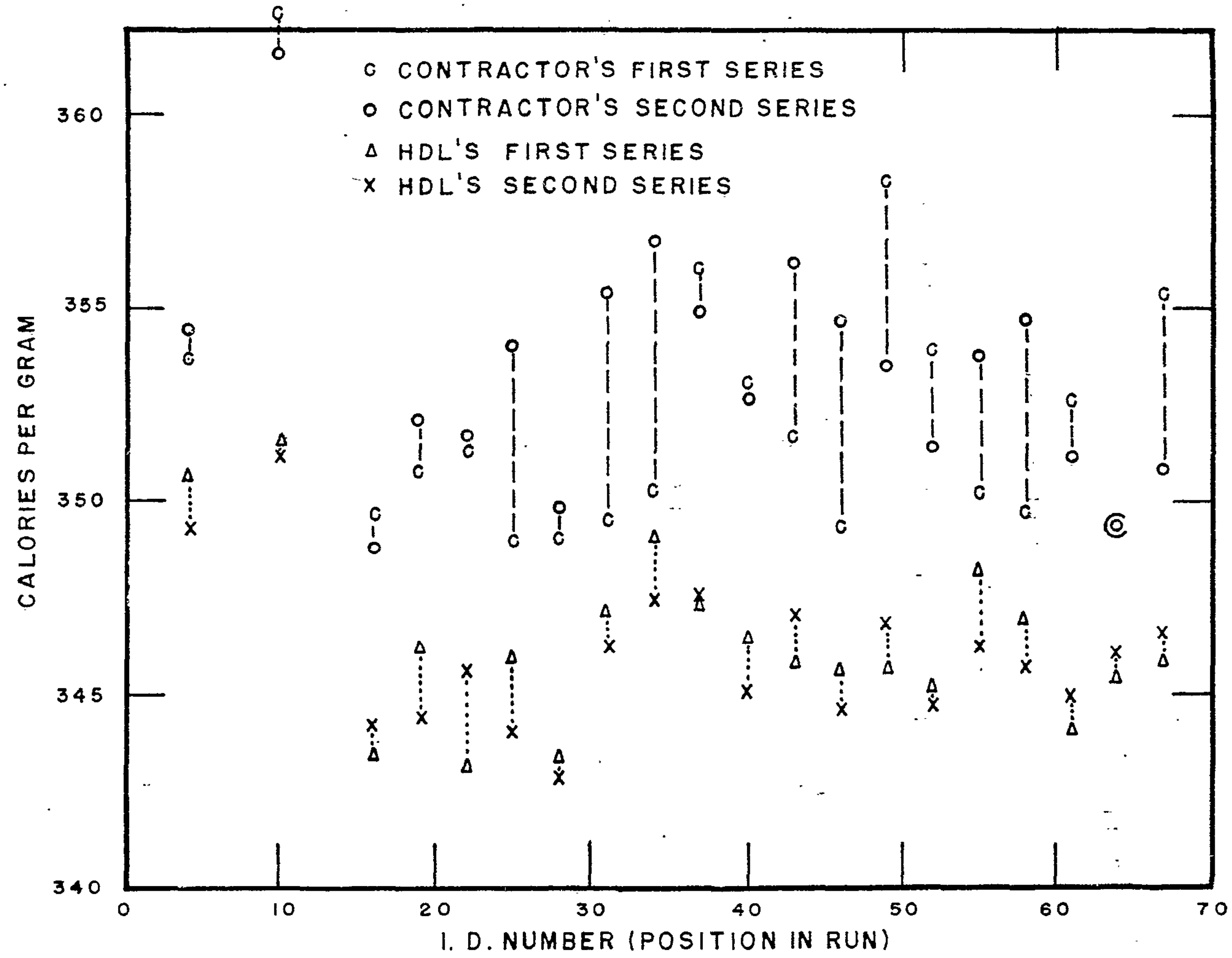

Figure 1. HDL and contractor calorimetric data for continuous-production heat-source material.

TABLE V. CALORIMETRIC DETERMINATIONS OF CONTINUOUS-PRODUCTION HEAT-SOURCE MATERIAL BY HDL AND BY THE CONTRACTOR

I.D. No.	Sample weight (g)		Total calorific value (cal)		Calorific value per unit weight (cal/g)	
	C	D	C	D	C	D
			SERIES No. 1			
4-1	3.6294	3.6057	1283.39	1264.40	353.61	350.67
10-1	3.6914	3.6492	1338.06	1282.96	362.48	351.57
16-1	3.6514	3.6738	1277.14	1261.82	349.77	343.46
19-1	3.6980	3.6889	1296.93	1277.29	350.71	346.25
22-1	3.6858	3.6855	1294.84	1265.43	351.31	343.25
25-1	3.7290	3.7129	1300.57	1284.51	348.99	345.96
28-1	3.4423	3.4332	1204.24	1177.24	349.84	342.90
31-1	3.5629	3.5618	1245.18	1237.55	349.48	347.17
34-1	3.6750	3.6750	1298.44	1277.29	353.67	347.56
37-1	3.5729	3.5807	1271.93	1243.77	355.94	347.35
40-1	3.5822	3.5870	1264.64	1242.74	353.04	346.46
43-1	3.7122	3.7230	1305.78	1287.60	351.75	345.85
46-1	3.4984	3.5035	1222.42	1212.41	349.42	345.73
49-1	3.5322	3.5293	1265.68	1220.56	358.33	345.84
52-1	3.6734	3.6781	1300.57	1273.68	354.05	346.29
55-1	3.5268	3.5311	1234.96	1229.85	350.17	348.29
58-1	3.5234	3.5326	1232.88	1226.24	349.91	347.11
61-1	3.5505	3.5660	1252.15	1227.78	352.67	344.30
64-1	3.5563	3.5555	1243.11	1228.81	349.55	345.61
67-1	3.5614	3.5806	1269.48	1239.13	356.46	346.07
			SERIES No. 2			
4-2	3.6112	3.6113	1279.74	1261.30	354.38	349.26
10-2	3.6511	3.6452	1319.84	1279.87	361.49	351.11
16-2	3.6591	3.6807	1272.45	1266.98	347.75	344.22
19-2	3.6877	3.6909	1298.48	1271.10	352.11	344.39
22-2	3.6912	3.6741	1306.88	1263.88	354.05	343.40
25-2	3.6980	3.7105	1290.68	1282.45	349.02	345.63
28-2	3.4313	3.4354	1206.79	1181.89	351.90	344.03
31-2	3.5500	3.5617	1261.63	1233.55	355.39	346.32
34-2	3.6651	3.6853	1307.41	1286.57	356.72	349.11
37-2	3.5775	3.5831	1269.61	1245.32	354.89	347.55
40-2	3.5825	3.5896	1263.60	1238.61	352.72	345.06
43-2	3.6997	3.7296	1321.78	1291.21	357.27	347.14
46-2	3.4822	3.5032	1238.74	1207.67	355.73	344.73
49-2	3.5225	3.5128	1245.66	1219.02	353.63	347.02
52-2	3.6715	3.6613	1290.68	1264.91	351.54	345.81
55-2	3.5164	3.5328	1244.06	1223.66	353.78	346.37
58-2	3.5215	3.5244	1249.38	1219.02	354.79	345.88
61-2	3.5536	3.5594	1248.50	1228.30	351.33	345.09
64-2	3.5415	3.5353	1238.09	1224.17	349.59	346.27
67-2	3.5684	3.5704	1252.67	1238.10	351.04	346.77

Series No. 1 Estimates

$\bar{C}_1 = 352.558$ $\bar{D}_1 = 346.385$

$\hat{\sigma}^2_{C_1} = 12.518$ $\hat{\sigma}^2_{D_1} = 4.691$

Series No. 2 Estimates

$\bar{C}_2 = 353.456$ $\bar{D}_2 = 346.258$

$\hat{\sigma}^2_{C_2} = 9.991$ $\hat{\sigma}^2_{D_2} = 3.723$

Combined Series Nos. 1 and 2
Estimates

$\bar{C} = 353.007$ $\bar{D}_2 = 346.321$

$\hat{\sigma}^2_C = 11.243$ $\hat{\sigma}^2_D = 4.085$

TABLE VI. STATISTICAL SUMMARY OF CONTINUOUS-PRODUCTION OF HEAT-SOURCE DATA.

		CONTRACTOR DATA				HDL DATA				
		Sample average (cal/g)	Number samples 20	Sample variance			Sample average (cal/g)	Number samples	Sample variance	
Continuous-process heat-source material: Series 1	$\Sigma C_{i1}/20 = \bar{C}_1$	352.558	20	$\hat{\sigma}^2_{C_1}$	12.518	$\bar{D}_1$	346.385	20	$\hat{\sigma}^2_{D_1}$	4.691
Continuous-process heat-source material: Series 2	$\Sigma C_{i2}/20 = \bar{C}_2$	353.456	20	$\hat{\sigma}^2_{C_2}$	9.991	$\bar{D}_2$	346.258	20	$\hat{\sigma}^2_{D_2}$	3.723
Combined Series 1 and 2	$\frac{\Sigma C_{iL} + \Sigma C_{i2}}{40} = \bar{C}$	353.007	40	$\hat{\sigma}^2_{C}$	11.243	$\bar{D}$	346.321	40	$\hat{\sigma}^2_{D}$	4.101
Individual difference (Series 1 - Series 2) = δ	$\frac{\Sigma(C_{i1} - C_{i2})}{20} = \overline{\delta C}$	-0.8985	20 pr	$\hat{\sigma}^2_{\delta C}$	11.285	$\overline{\delta D}$	0.1315	20 pr	$\hat{\sigma}^2_{\delta D}$	1.233
Homogeneous heat powder Series 1	$\Sigma C^*_{i1}/20 = \bar{C}^*_1$	348.538	20	$\hat{\sigma}^2_{C^*_1}$	1.869	$\bar{D}^*$	342.646	20	$\hat{\sigma}^2_{D^*}$	0.071
Homogeneous heat powder* Series 2	$\Sigma C^*_{i2}/20 = \bar{C}^*_2$	347.906	20	$\hat{\sigma}^2_{C^*_2}$	3.801					
Combined series 1 and 2 Homogeneous heat powder	$\frac{\Sigma C^*_{i1} + \Sigma C^*_{i2}}{40} = \bar{C}^*$	348.220	40	$\hat{\sigma}^2_{C^*}$	2.860					

* Second series not required at HDL

The subscripts 1 and 2 indicate the series of data from which the particular statistic was computed. The symbol δ refers to a difference between values, and $\overline{\delta D}$ and $\overline{\delta C}$ are the averages of differences for data obtained by HDL and by the contractor, respectively. The letter N refers to the number of observations, and a subscript to the letter N shows the source of observations. MSstr and MSadj refer to the mean squares of the determinations.

In all the test work, the unit of measurement was calories per gram; however, the computed test statistic is dimensionless.

3.1 Equality of Calorific Value for Adjacent Samples

The averages of the differences in calorific values between adjacent sections of continuous-production heat-source material are $\overline{\delta D}$ and $\overline{\delta C}$, for HDL and the contractor, respectively. If they do not differ significantly from zero, then it can be inferred that the values for the adjacent parts are equivalent. To test for the significance, student's t must be computed:

$$t = \frac{\overline{\delta D}}{\sqrt{\hat{\sigma}^2_{\delta D}/N}} = \frac{0.1315}{\sqrt{1.233/20}} = .530$$

$$t = \frac{\overline{\delta C}}{\sqrt{\hat{\sigma}^2_{\delta C}/N}} = \frac{-0.898}{\sqrt{11.285/20}} = -1.195$$

These values are compared with the critical value of $t_{.01} = 2.86$ (ref 3) for twenty observations in each sample. Since the critical value is not exceeded, it can be concluded that the average calorific values for adjacent parts are equal.

3.2 Variance in Calorific Value of Continuous-Production Heat-Source Material Compared with Homogeneous Heat-Source Material

The inequalities $\sigma^2_D > \sigma^2_{D*}$ and $\sigma^2_C > \sigma^2_{C*}$ state that the true variances of the calorific values for continuous-production material exceed those for the homogeneous material, both at HDL and at the contractor's laboratory. To test for this condition, the quantity F is computed:

$$F = \hat{\sigma}^2_D/\hat{\sigma}^2_{D*} = 4.085/.071 = 57.5$$

For $N_D = 40$, and for $N_{D*} = 20$, the critical value (ref 3) is $F_{.01} = 2.76$

$$F = \hat{\sigma}^2_C/\hat{\sigma}^2_{C*} = 11.243/2.860 = 3.93$$

For $N_C = N_{C*} = 40$, the critical value (ref. 3) is $F_{.01} = 2.14$

In both instances, the computed values exceed the critical values and it can be concluded that the continuous-production heat-source material is significantly more variable than the homogeneous heat powder, a result to be expected. These two tests provide comparisons of the measurement or instrument variance with the variance due to the material itself. The latter contains the variance that accounts for the small F ratio from the contractor's measurements. Using the HDL data to estimate the precision of the measurements, the attained variance ratio of about 60:1, in addition to the small coefficient of variation indicates that the Parr Instrument Company Model 1411 calorimeter is a satisfactory device for the measurement of the heat evolved from the burning of the continuous-production heat-source-material.

3.3 Effect of Scrutinizing the Calorimetry

In Table V the contractor's first set of observations C_1 was made on the regular quality control sample of continuous-production material, and the second set of observations, C_2, was made on the adjacent samples. These latter samples were assigned random code numbers and given to a technician to evaluate. The technician realized that this procedure was not a routine measurement of the production material; it might be conjectured that the latter samples would receive special attention, and the resulting measurements would evidence increased precision, i.e., decreased variance. The computed values of $\hat{\sigma}^2_C$ and $\hat{\sigma}^2_{C_2}$ on the continuous-production material were used to make this comparison. From the point of view of lower variability it would have been preferable to have based this test on the homogeneous heat powder. However, all homogeneous heat powder runs were special, and no "routine" data were obtained that could be used to make this test.

Based on the data in Table V, if $\sigma_{C_1} > \sigma_{C_2}$ it can be concluded that, under the conditions of the experiment, scrutiny results in less variability; conversely, if $\sigma_{C_1} < \sigma_{C_2}$, then that conclusion cannot be drawn. To make the test, the quantity

$$F = \hat{\sigma}^2_{C_1} / \hat{\sigma}^2_{C_2} = 12.518/9.991 = 1.252$$

is computed. The critical value for $N_{C_1} = N_{C_2} = 20$ is $F_{.01} = 3.03$. This value is not exceeded by the computed value of F; hence, it cannot be concluded that C_2 is less variable than C_1, and it must be assumed that scrutiny does not affect variability.

3.4 Calorific Variation in Continuous-Production Heat-Source Material

Table VII presents an analysis of stream variance in the continuous-production material as determined by calorimetric measurements of HDL and the contractor. In drawing conclusions with respect to stream variation,

TABLE VII. ANALYSIS OF VARIANCE OF CONTINUOUS-PRODUCTION HEAT-SOURCE MATERIAL

Source of Variation	HDL DATA Sum of squares	HDL DATA Degrees of freedom	HDL DATA Mean square (MS)	CONTRACTOR DATA Sum of squares	CONTRACTOR DATA Degrees of freedom	CONTRACTOR DATA Mean square (MS)
Downstream[1] (str)	147.43	19	7.759	323.14	19	17.007
Adjacent pads[2] (adj)	11.90	20	0.595	115.34	20	5.567
Total[3]	159.33	39	4.085	438.48	39	11.243
Homogeneous heat powder[4] (measurement error)		19	0.071		39	2.860

[1] This between-variation is used to estimate the variation of the average of the pairs about the grand average.

[2] This within-variation is a measure of the variation of the individuals in a pair of observations about the respective pair average.

[3] The total is the same as $\hat{\sigma}^2_D$ and $\hat{\sigma}^2_C$ in Table VI. This is a measure of the variation of the individual observations about the grand mean.

[4] This is the same as $\hat{\sigma}^2_{D*}$ and $\hat{\sigma}^2_{C*}$ in Table VI. The homogeneous heat-powder variance is used for the external estimate of the variance due to measurement.

the HDL rather than the contractor's measurements will be used because they are more precise and any statistical tests with them will be more sensitive; in addition, their distributions are closer to normal so that the validity of the inferences is strengthened.

In Table VII the total variation is equivalent to the combined variation of series 1 and 2, also shown in Table V; it is a measure of the variation of the individual observations about the combined average. The downstream variation, also called the between-sum-squares, is used to measure the variation of the average of the two adjacent observations about the combined average. The adjacent-pad mean sum-of-squares, also called the within-variance, is a measure of the variation of the twenty pairs of observations about their respective averages. The variance in the homogeneous heat powder is taken from Table VI and will serve as an estimate of the measurement variance, or instrument variance, and hence, of the imprecision of the instrument; the square root of its reciprocal is a measure of the precision.

3.4.1 Comparison of the Downstream and the Adjacent-pad Variations with the Variation due to Measurement Alone

If MSstr is significantly greater than $\hat{\sigma}^2_{D*}$, and MSadj is significantly greater than $\hat{\sigma}^2_{D*}$, then the inference is that neither the adjacent-pad variation nor the downstream variation can be attributed to calorimetry alone.

$$F = \widehat{MSadj}/\hat{\sigma}^2_{D*} = 0.5950/0.071 = 8.380$$

The critical value (ref 3) is $F_{.01} = 3.00$

$$F = \widehat{MSstr}/\hat{\sigma}^2_{D*} = 7.759/0.71 = 109.28$$

The critical value (ref 3) is $F_{.01} = 3.03$

In both instances the computed values far exceed the critical value; thus, the inferences follow. Neither the variation between adjacent pads nor the downstream variation can be attributed to calorimetry. Hence, there are both short-term and long-term fluctuations during a production run.

3.4.2 Comparison of Downstream Variation with Adjacent-Pad Variation

The quantity $F = \widehat{MSstr}/\widehat{MSadj} = 7.759/0.595 = 13.04$ is computed and compared with the critical value $F_{.01} = 2.96$ (ref 3). Since the computed F exceeds the critical value, it is concluded that the downstream variation is significantly larger than the adjacent-pad variation. Hence the long-term fluctuations in production are more deleterious than the short-term ones.

3.4.3 Comparison of Contractor's Downstream and Adjacent-Pad Variances with Those of HDL

Although the contractor's data were not used for making the previous two statistical tests, the mean sums-of-squares from his data can be examined and compared with those of HDL. The mean squares can be treated as variances, except that the downstream component must be reduced by twice the amount of the adjacent-pad MS. The comparison shows that the estimate of the downstream components of variance for HDL and for the contractor are about equal, but the contractor's adjacent-pad variances are larger than those of HDL, as are his homogeneous heat-powder variances.

4. CONCLUSIONS

When estimates of precision are expressed as a percent of mean, it is found that the precision of determinations using benzoic acid is almost identical with the precision using homogeneous heat powder. When the total calories is the same and the estimates of precision are expressed in units of calories per gram, use of the homogeneous heat powder results in greater precision than does use of benzoic acid, because the effect of weighing errors is increased. Since the benzoic acid can also be used to check accuracy of the instrument, however, it is the preferred material for calibration.

Using homogeneous heat powder, the variance in measurements by HDL was 0.071, for the contractor, 2.86; and these differences were shown to be statistically significant. A positive bias of 5.85 cal/g was found in the contractor's measurements. A recommendation that the contractor modify procedures to match the HDL procedures to reduce variance and bias, has been accepted.

Analysis of calorimetric data from a continuous-production run of the contractor's heat-source material showed this material is significantly more variable than the previously mentioned homogeneous heat powder. In addition, significant short-term, and larger long-term, fluctuations in the manufacturing process were indicated. A change in the manufacturing process would be required if the long-term fluctuations are to be reduced. More precise measurements would reduce the contractor's short-term fluctuations.

Since the calorimeter variance is so much smaller than the variance of the production-run heat material, the Parr Model 1411 calorimeter is suitable for measuring the heat evolved from heat-source materials provided that the measurements are carefully made.

5. REFERENCES AND BIBLIOGRAPHY

(1) Ira R. Marcus, DOFL Report No. TR-576, "Measurement of Heat Evolved by Thermite Mixtures, Part III, Standard Calorimetry for Quality Control," 15 January 1958.

(2) Mardelle L. Couch and Ira R. Marcus, DOFL Report No. R-320-59-12, "Calibration of Parr No. 1411 Heat Powder Calorimeter."

(3) A. H. Bowker and G. J. Lieberman, "Handbook of Industrial Statistics," p 827, Prentice-Hall, Inc., N. J., 1956.

(4) R. H. Comyn and I. R. Marcus, DOFL Report No. TR-862, "Heat Powder Calorimetry," 1 August 1960.

ACKNOWLEDGMENT

The authors wish to express their appreciation to Mr. Lloyd Berry for his carefully done calorimetry and for the assistance of Miss Eleanor F. Horsey in the preparation of this report.

DISTRIBUTION

Commanding General
U.S. Army Materiel Command
Washington 25, D. C.
    Attn: AMC-RD-DE-N

Redstone Scientific Information Center
U. S. Army Missile Command
Redstone Arsenal, Alabama
    Attn: Chief, Document Section
    Attn: Hugh Camp (R & D Div)

Commanding General
U. S. Army Munitions Command
Dover, New Jersey
    Attn: Technical Documents Library
    Attn: FREL—Pyrotechnics Laboratory

Commanding Officer
U. S. Army Research Office (Durham)
Box CM, Duke Station
Durham, North Carolina
    Attn: CRD-AA-IP

Commander
U. S. Naval Ordnance Laboratory
Corona, California
    Attn: Charles Haber
    Attn: Documents Librarian

Commander
U. S. Naval Ordnance Laboratory
White Oak, Silver Spring 19, Maryland
    Attn: A. G. Hellfritzsch
    Attn: R. C. Daniel
    Attn: Technical Library
    Attn: A. Lightbody
    Attn: I. D. Yalom
    Attn: Bernard White, Chemical Engineering Dept.

U. S. Naval Ammunition Depot
Crane, Indiana
    Attn: V. C. Willis

Commander
Naval Research Laboratory
Washington 25, D. C.
    Attn: Tech Library

Commander
U. S. Naval Ordnance Test Station
China Lake, California
Attn: Tech Library
Attn: Jack Sherman, Salt Wells Station

Commander
Armed Services Technical Information Agency
Arlington Hall Station
Arlington 12, Virginia
Attn: TIPDR (10 copies)

U. S. Atomic Energy Commission
Washington 25, D. C.
Attn: Technical Reports Library

Jet Propulsion Laboratory
Pasadena, California
Attn: Library

Catalyst Research Corporation
6101 Falls Road
Baltimore 9, Maryland
Attn: Elmer McKee
Attn: Quality Control Dept

Eagle Picher Company
P. O. Box 290
Joplin, Missouri
Attn: M. F. Chubb

Eureka-Williams Company
East Bell Street
Bloomington, Illinois
Attn: Lyle Waller

Minneapolis-Honeywell Regulator Company
600 Second Street, N.
Hopkins, Minnesota
Attn: Paul Joyner

Pass & Seymour, Inc
Solvay Station
Syracuse, New York
Attn: C. H. H. Roberts

Sandia Corporation
Sandia Base
Albuquerque, New Mexico
Attn: F. A. Goss (3 copies)

DISTRIBUTION (Continued)

Universal Match Corporation
P. O. Box 191
Ferguson 21, Missouri

Universal Match Corporation
P. O. Box 5841
Ferguson 35, Missouri
Attn: Dr. H. Ellern

Universal Match Corporation
P. O. Box 231
Marion, Illinois
Attn: Samuel M. Carter

The Wurlitzer Company
North Tonawanda, New York
Attn: William O'Hearn

University of Florida
P. O. Box 3027, University Station
Gainesville, Florida
Attn: R. D. Walker

Internal

Horton, B. M./McEvoy, R. W., Lt Col
Apstein, M./Gerwin, H. L./Guarino, P.A./Kalmus, H. P.
Spates, J. E./Schwenk, C. C.
Hardin, C. D., Lab 100
Sommer, H., Lab 200
Hatcher, R. D., Lab 300
Hoff, Robert S., Lab 400
Nilson, H. M., Lab 500
Flyer, I. N., Lab 600
Campagna, J. H./Apolenis, C. J., Div 700
DeMasi, R., Div 800
Landis, P. E., Lab 900
Seaton, J. W., 260
Piper, W., 450
Goodrich, R. B., 940 (3 copies)
Marcus, I. R., 450
Comyn, R. H., 450 (5 copies)
Lipnick, M., 710 (15 copies)
Davis, B. L., 940
Levin, S. G., 900 (10 copies)
Keehn G. R., 710
Hens, R. J., 710 (5 copies)
Technical Reports Unit, 800
Technical Information Office, 010 (10 copies)
HDL Library (5 copies)
Rotkin, I/Godfrey, T. B./Eichberg, R. L.
Bryant, W. T./Distad, M. F./McCoskey, R. E./Moorhead, J. G.

AD____________ Accession No.__________

Harry Diamond Laboratories, Washington 25, D. C.

STATISTICAL EVALUATION OF CALORIMETRIC MEASUREMENTS ON HEAT-SOURCE MATERIALS--Sheldon G. Levin and Robert E. McIntyre

Calorimeters

TR-1028, 28 March 1963, 19 pp text, 1 illus., OMS Code 4070.17.30021 HDL Proj 75333, UNCLASSIFIED Report

A statistical study was made to determine the precision of calorimetric determinations made on the Parr Model 1411 calorimeter. In addition, the calorific values for continuous-production heat-source material and for an experimentally prepared homogeneous material were determined at Harry Diamond Laboratories (HDL) and by a contractor, and the variability of measurements are compared. It was found that operator-skill and techniques employed can result in a significant increase in the precision of measurements obtained. HDL procedures intended to increase precision are given.

When the determinations with the Parr Model 1411 calorimeter are carefully made, the variance in calorific values due to the calorimetry is small compared to the variance of the determinations made on samples from a typical run of continuous-production heat-source material.

AD____________ Accession No.__________

Harry Diamond Laboratories, Washington 25, D. C.

STATISTICAL EVALUATION OF CALORIMETRIC MEASUREMENTS ON HEAT-SOURCE MATERIALS--Sheldon G. Levin and Robert E. McIntyre

Calorimeters

TR-1028, 28 March 1963, 19 pp text, 1 illus., OMS Code 4070.17.30021 HDL Proj 75333, UNCLASSIFIED Report

A statistical study was made to determine the precision of calorimetric determinations made on the Parr Model 1411 calorimeter. In addition, the calorific values for continuous-production heat-source material and for an experimentally prepared homogeneous material were determined at Harry Diamond Laboratories (HDL) and by a contractor, and the variability of measurements are compared. It was found that operator-skill and techniques employed can result in a significant increase in the precision of measurements obtained. HDL procedures intended to increase precision are given.

When the determinations with the Parr Model 1411 calorimeter are carefully made, the variance in calorific values due to the calorimetry is small compared to the variance of the determinations made on samples from a typical run of continuous-production heat-source material.

AD____________ Accession No.__________

Harry Diamond Laboratories, Washington 25, D. C.

STATISTICAL EVALUATION OF CALORIMETRIC MEASUREMENTS ON HEAT-SOURCE MATERIALS--Sheldon G. Levin and Robert E. McIntyre

Calorimeters

TR-1028, 28 March 1963, 19 pp text, 1 illus., OMS Code 4070.17.30021 HDL Proj 75333, UNCLASSIFIED Report

A statistical study was made to determine the precision of calorimetric determinations made on the Parr Model 1411 calorimeter. In addition, the calorific values for continuous-production heat-source material and for an experimentally prepared homogeneous material were determined at Harry Diamond Laboratories (HDL) and by a contractor, and the variability of measurements are compared. It was found that operator-skill and techniques employed can result in a significant increase in the precision of measurements obtained. HDL procedures intended to increase precision are given.

When the determinations with the Parr Model 1411 calorimeter are carefully made, the variance in calorific values due to the calorimetry is small compared to the variance of the determinations made on samples from a typical run of continuous-production heat-source material.

AD____________ Accession No.__________

Harry Diamond Laboratories, Washington 25, D. C.

STATISTICAL EVALUATION OF CALORIMETRIC MEASUREMENTS ON HEAT-SOURCE MATERIALS--Sheldon G. Levin and Robert E. McIntyre

Calorimeters

TR-1028, 28 March 1963, 19 pp text, 1 illus., OMS Code 4070.17.30021 HDL Proj 75333, UNCLASSIFIED Report

A statistical study was made to determine the precision of calorimetric determinations made on the Parr Model 1411 calorimeter. In addition, the calorific values for continuous-production heat-source material and for an experimentally prepared homogeneous material were determined at Harry Diamond Laboratories (HDL) and by a contractor, and the variability of measurements are compared. It was found that operator-skill and techniques employed can result in a significant increase in the precision of measurements obtained. HDL procedures intended to increase precision are given.

When the determinations with the Parr Model 1411 calorimeter are carefully made, the variance in calorific values due to the calorimetry is small compared to the variance of the determinations made on samples from a typical run of continuous-production heat-source material.

REMOVAL OF EACH CARD WILL BE NOTED ON INSIDE BACK COVER, AND REMOVED CARDS WILL BE TREATED AS REQUIRED BY THEIR SECURITY CLASSIFICATION.